Radiation Damage in Polarized Ammonia Solids

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Abstract. Solid $^{15}NH_3$ and $^{15}ND_3$ provide a highly polarizable, radiation resistant source of polarized protons and deuterons and have been used extensively in high luminosity (10^{35} cm^{-2}s$^-$1) experiments investigating the spin structure of the nucleon. Over the past twenty years, the UVA polarized target group has been instrumental in producing and polarizing much of the material used in these studies, and many practical considerations have been learned in this time. In this discussion, we analyze the polarization performance of the solid ammonia targets used during the recent JLab Eg4 run. Topics include the rate of polarization decay with accumulated charge, the annealing procedure for radiation damaged targets to recover polarization, and the radiation induced change in optimum microwave frequency used to polarize the sample. We also discuss the success we have had in implementing frequency modulation of the polarizing microwave frequency.

Keywords: Polarized Ammonia, $^{15}NH_3$, $^{15}ND_3$, Radiation Damage, Annealing
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INTRODUCTION

The Jefferson Lab Eg4 [1] experiment ran in 2006 with the goal of extracting the proton and deuteron g_1 structure functions, and the extended GDH Sum at low Q^2. For this purpose we utilized the JLab Hall B polarized target apparatus [2] loaded with solid $^{15}NH_3$ and $^{15}ND_3$. We discuss here some phenomenological observations regarding the polarized target material used during the experiment. These observations are of a practical nature and are in general consistent with previous experience [3, 4].

The target's 5 Tesla magnetic field is produced by a split Helmholtz pair superconducting magnet. And the cooling power needed to handle the heat deposited by the JLab electron beam is supplied by a 1 Kelvin ^{4}He evaporation refrigerator. Approximately, 1 Watt of microwave power at 140 GHz is used to pump electrons during the process of Dynamic Nuclear Polarization (DNP). Complete details of the apparatus and polarization procedure can be found in Ref. [2].

RADIATION DAMAGE

To create the paramagnetic centers that are needed for the DNP process, raw frozen ammonia must be irradiated with a dose of approximately 10^{17} e$^-$/cm^2. This *pre-irradiation* was performed with the material at 87 K under liquid Argon at a low energy electron accelerator prior to the start of the experiment. With this treatment, $^{15}NH_3$ typically polarizes to greater than 90% at 5 T and 1 K on the initial spinup.
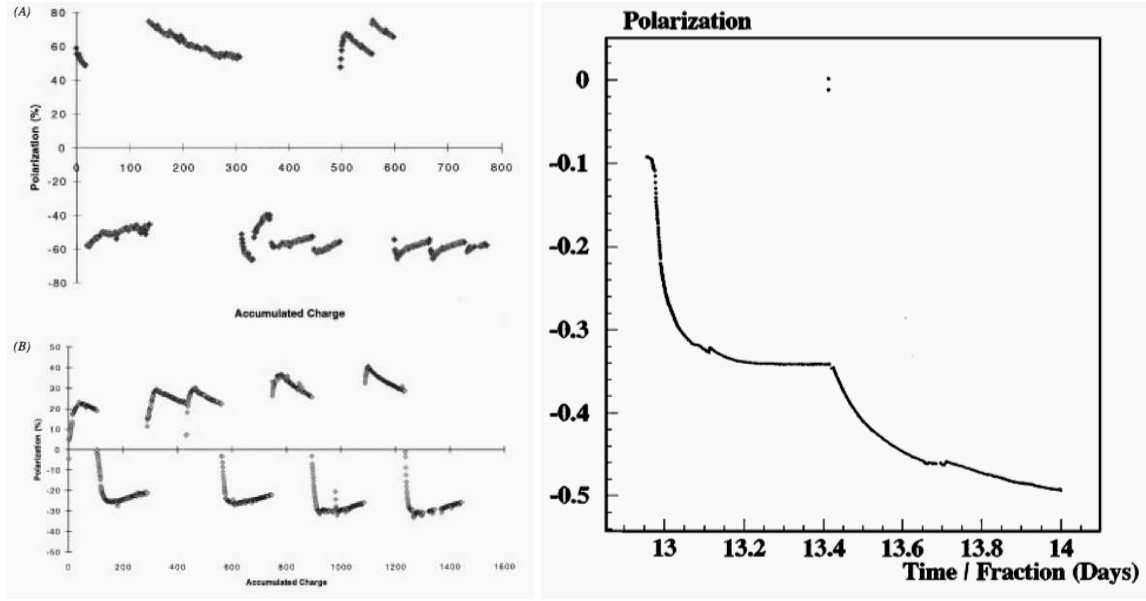
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[1] On behalf of the UVA Polarized Target Group.

FIGURE 1. **Left:** Typical behaviour of $^{15}NH_3$ (top) and $^{15}ND_3$ (bottom) with accumulated dose. **Right:** The effect of applying microwave frequency modulation to deuterated butanol during the SMC [6, 7, 8] experiment. Frequency Modulation applied at $T \approx 13.4$. Both figures reproduced with permission from [5].

$^{15}ND_3$ behaves quite differently, as displayed in the lower left panel of Fig. 1. The initial deuteron polarization is typically less than 20%, but further *cold irradiation* with the target in beam at 1 K subsequently increases the maximum $^{15}ND_3$ polarization to 40-45% (5T/1K) after an additional cold dose of about 10^{16} e$^-$/cm^2.

Radiation damage is the creation of unwanted radicals in the material which do not participate in the DNP process, but which do provide an additional relaxation mechanism. As the dose accumulates, the polarization decays in a roughly exponential fashion. When the polarization falls to unacceptable levels, the target is subjected to annealing. This is the process of reducing the concentration of these unwanted radicals in the material by warming the material to temperatures similar to the conditions of pre-irradiation. Typically this process restores the maximum achievable proton polarization to the previous best value, and can be repeated many times before the material needs to be replaced. The deuteron on the other hand does not reach it's maximum polarization until it has been subjected to several dosing and anneal cycles. There is of course some risk involved with every anneal, since it is possible to destroy the paramagnetic centers needed for DNP if the temperature is raised too high. During Eg4 we annealed at approximately 90K for 1 hour each time with good results.

The Eg4 beam current was limited to a few nanoAmps due to the luminosity limits of the Hall B detector package. This is about two orders of magnitude less intense than was used in the previous Hall C and SLAC polarized target experiments. To achieve sufficient target dosing, it was necessary to periodically disable the detector stack and irradiate the target with 100nA beam. We scheduled these one hour cold dose runs as frequently as possible during the run. The improvement in polarization (and figure of merit, which
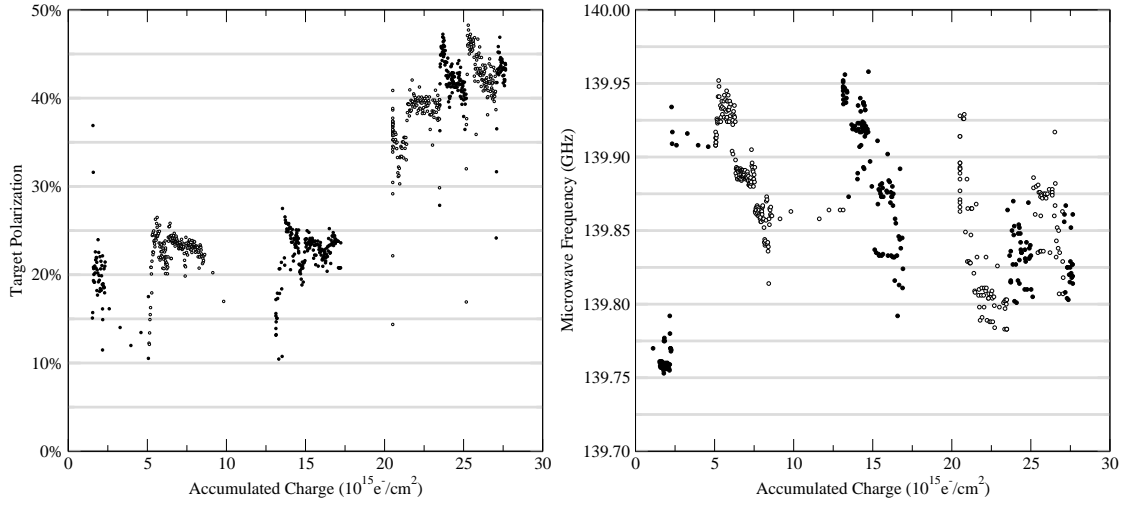
FIGURE 2. **Left:** Deuteron polarization as a function of accumulated dose. Note that Eg4 polarized $^{15}ND_3$ only in the positive state unlike what is shown in Fig. 1. **Right:** Eg4 Deuteron optimal microwave frequency as a function of accumulated dose. Open/closed symbols are used to indicate different anneal cycles.

depends on the polarization squared) is reflected in Fig. 2 and was substantial.

The Eg4 program was temporarily interupted by a failure of the JLab End Station Refrigerator (ESR) which provides liquid cryogenic to the Halls. During this time it was necessary to remove the $^{15}ND_3$ from the target and store the material under liquid nitrogen for several days while the ESR recovered. This had the effect of providing an extended, low level anneal of the material. When the $^{15}ND_3$ was replaced and subjected to a further cold dose, the polarization grew by 10% as shown in Fig. 2, and continued to grow with further dose. At the end of the run, the deuteron polarization had increased to greater than 45%. During this time the material had absorbed less than 30 x 10^{15} e^-/cm^2. Previous experience [3, 4] has shown that ND_3 is extremely radiation hard, able to withstand up to 100 x 10^{15} e^-/cm^2 before replacement is needed.

DNP MICROWAVE FREQUENCY

There are two possible DNP pumping frequencies depending on the energy transition selected. The initial separation of the two frequencies is about 200 MHz, but these optimal frequencies drift steadily apart with accumulated dose reflecting the change in material caused by the radiation. The approximate 100 MHz variation in optimum frequency over each anneal cyle is shown in the right panel of Fig. 2 for the $^{15}ND_3$ target. Over the first few cycles the optimal frequency dropped sharply with dose, and returned to it's previous initial value after an anneal. This behaviour is not as apparent after a dose of about $20 \cdot 10^{16}$ e^-/cm^2, during the high polarization part of Eg4. The polarization frequency was adjusted by hand and reflects the judgement of the target operator as to what is optimal at that moment, so in this regard, it is not clear whether the increased scatter in optimal frequency during the latter part of the experiment reflects
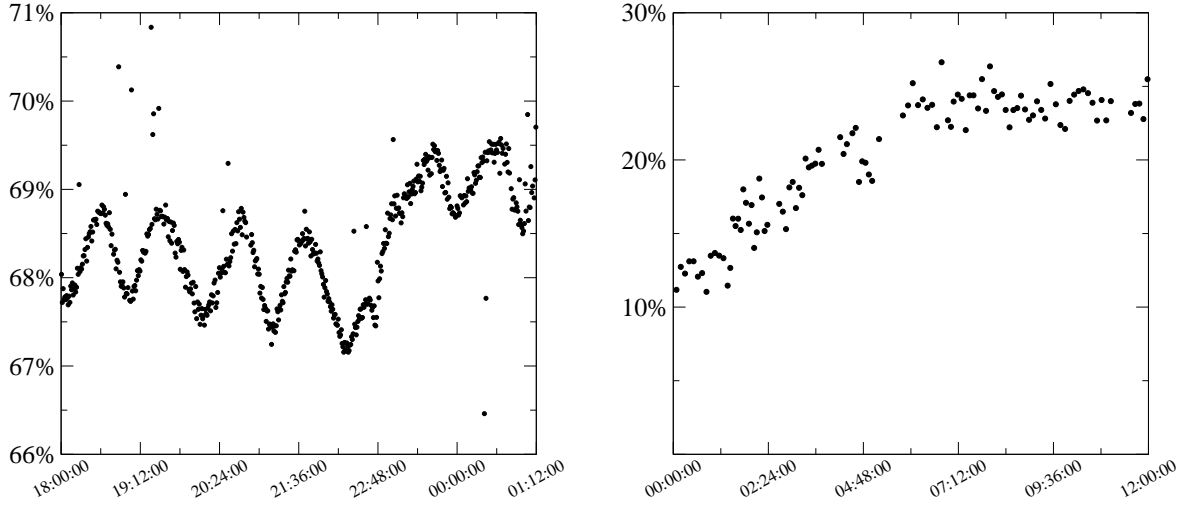
FIGURE 3. Polarization vs. Time showing effect of the application of Frequency Modulation to microwaves. **Left:** Proton polarization. FM activated at approximately 22:48. Note the sinusoidal background is caused by mistuned cryogenic PID loop and is unrelated to the frequency modulation. **Right:** Deuteron polarization. FM activated at approximately 04:48.

an actual change in material characteristics as might also be inferred from the figure.

Frequency modulation of microwave frequency has been shown to have a dramatic effect on the maximum achievable polarization. See for example, Fig. 1 from Ref. [5]. With this in mind, we implemented a modulation of the polarizing microwave frequency during Eg4 using a 1 kHZ 5 Volt peak-to-peak signal. While not as dramatic as previously published results, we did observe a few percent improvement in polarization for both the proton and deuteron target polarization as shown in Fig. 3.

CONCLUSION

While $^{15}NH_3$ typically polarizes to greater than 90% after an initial warm irradiation of approximately 10^{17} e$^-$/cm^2 at 87 K, a further series of cold irradiations and anneals are crucial to obtain large polarizations in $^{15}ND_3$. Annealing for 90-100K for approximately one hour after the polarization has dropped brings proton polarization back to previous maximum, while it typically increases the maximum possible polarization in $^{15}ND_3$.

This target technology will be used again in 2008 for the SANE [9], SemiSANE [10], Wide Angle Compton Scattering [11], and g_1^D/F_1^D [12] experiments in Jefferson Lab Hall C. In addition, the δ_{LT} experiment [13] which will measure the proton spin structure function g_2^p and generalized spin polarizability δ_{LT} will require a first time installation of this target in Jefferson Lab Hall A.

ACKNOWLEDGMENTS

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